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A simple stir casting technique for the preparation of *in situ* Fe-aluminides reinforced Al-matrix composites☆

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Summary This article presents a simple stir casting technique for the development of Fe-aluminides particulate reinforced Al-matrix composites. It has been demonstrated that stirring of super-heated Al-melt by a mild steel plate followed by conventional casting and hot rolled results in uniform dispersion of *in situ* $Al_{13}Fe_4$ particles in the Al matrix; the amount of reinforcement is found to increase with increasing melt temperature. With reference to base alloy, the developed composite exhibits higher hardness and improved tensile strength without much loss of ductility; since, composite like base alloy undergoes ductile mode of fracture.

Introduction

Discontinuous particulate reinforced Al-matrix composites (AMCs) have received increasing application in aerospace and automobile sectors due to their superior strength-to-weight ratio, not easily attainable in monolithic alloys, as well as higher strength-to-cost ratio, relative ease of fabrication and isotropic properties as compared to other varieties of composites (Ibrahim et al., 1991). Compared to solid-phase processing, the liquid-phase processing methods are obviously attractive as they are economical and also capable to produce large structural components with complex geometry. However, major challenge in the liquid phase processing is to achieve uniform distribution of reinforcement and to obtain strong interfacial bonding between the reinforcement and the matrix (Hashim et al., 2002). Degrees of success in both of these aspects are limited, because the commonly employed reinforcements in AMCs are ceramics that exhibit poor wettability in the liquid-Al, and due to the fact that the final product is a mixture of

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essentially two completely dissimilar materials (Surappa, 2003). Some of these problems could be alleviated if the reinforcement would coexist in a more-or-less equilibrium state with the Al-matrix (Tjong and Ma, 2000). Therefore, transition metal tri-aluminides intermetallic have attracted considerable attention in recent years as reinforcement in the AMCs, because they possess lower density, higher hardness and strength even at elevated temperature due to their ordered structure (Varin, 2002). Furthermore, tri-aluminides can be formed with relative ease as compared to ceramic particles via *in situ* reaction between the matrix and the precursor element/compound during fabrication, having obvious advantages over *ex situ* technique like better control over size and distribution of reinforcements as well as the characteristics of the particle-matrix interface resulting in much superior mechanical property (Lee et al., 2003). The present article reports a simple stir casting technique for bulk production of *in situ* Fe-aluminides reinforced AMCs.

Experimental procedures

Commercially pure Al (Al-0.08Fe-0.06Si, all in wt.%) and 2 wt.% Mg were melted in an alumina crucible inside an electrically heated pot furnace at 760 °C. The liquid metal was then mechanically stirred using graphite stirrer for preparation of base alloy. For the preparation of Fe-aluminides reinforced AMC, a sacrificial type mild steel (Fe-0.29C-0.05Mn-0.2Si, all in wt.%) plate (165 mm × 18 mm × 6 mm) was used as a stirrer. The starrer itself acts as the source of Fe for the formation of Fe-aluminides. After preparation of Al-melt of nominal composition of Al-2Mg, the melt was heated to the pre-determined temperature. Subsequently, the mild steel plate attached with central stirring rod was dipped 60 mm in the liquid metal and kept static for about 1 min followed by mechanical stirring for 6 min at an average speed of 400 rpm. The molten-Al was reacted with the solid Fe of mild steel stirrer and Fe-aluminides intermetallic particles were generated. With a view to increase the amount of Fe-aluminides, the temperature of molten-Al was varied from 760 to 880 °C at uniform interval of 40 °C. After completion of stirring, a small tablet of hexachloroethane was added into the melt for degassing. The crucible was then manually taken out of the furnace and the molten alloy with the reaction product was poured into the preheated cast iron die (200 mm × 60 mm × 10 mm). As-cast materials were heated at 540 °C for 1 h followed by hot rolling to a final thinness of 7 mm. The hot rolled materials were then normalised at 400 °C for 1 h.

Following standard metallographic practices, the microstructural features of the developed materials were studied with the aid of optical (Carl Zeiss, Axiovert 40 MAT) and scanning electron microscope, SEM (JEOL, JSM 5510) equipped with energy dispersive X-ray (EDX) facility (NORAN, System six). The amount of Fe-aluminides was estimated using of SEM micrographs taken in the back scatter electron mode by image analyses technique with the help of Axiom-Vision 3.8.2 software. X-ray diffraction (XRD) patterns of all specimens were recorded to identify different phases present in the microstructures using a diffractometer (Bruker D8) with Cu-K_α radiation. Hardness measurements were carried out by using a Vickers microhardness tester (Leica, Micro system GmbH) with an applied load of 500 gf and dwell time of 15 s. Following ASTM E8M standard, tensile tests of developed materials were also carried out using flat specimens of 25 mm gauge length with the help of computer controlled universal testing machine (INSTRON, 8810) at room temperature and crosshead velocity of 0.50 mm min^{-1}. Fractured surface of broken tensile samples were examined under SEM.

Results and discussion

Microstructure

Fig. 1 depicts representative SEM micrograph of un-etched composite prepared at the melt temperature of 880 °C. Micrograph reveals uniform dispersion of intermetallic phase that appears white in the SEM micrograph taken in the back scatter mode (Fig. 1(a)). The results of EDX micro-analyses in Fig. 1(b) confirm that the white regions in the SEM micrograph are Fe-rich unlike black matrix. The intermetallic phase is primarily fibre shaped with typical length of 5—15 μm and thickness of 2—4 μm. The distribution of the intermetallic phase suggests that these are segregated in the inter-dendritic region during casting of the composite.

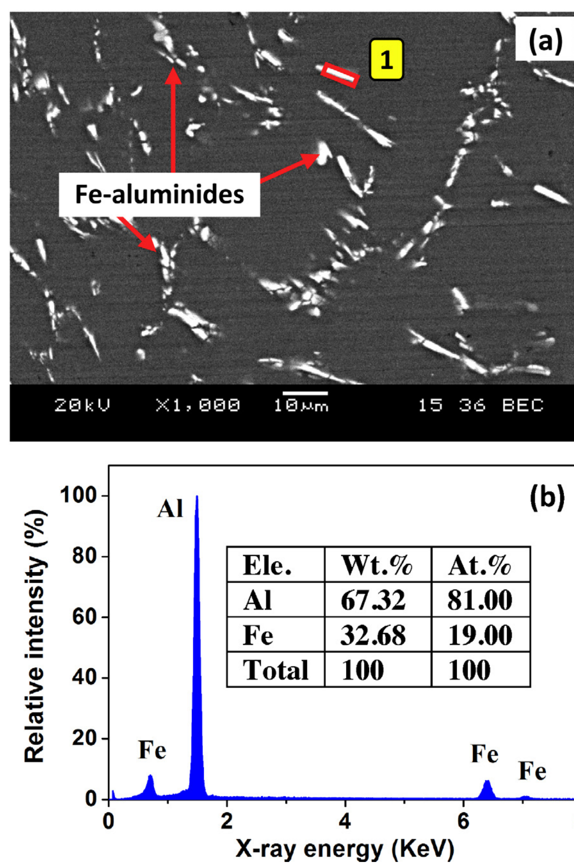

Figure 1 (a) Representative SEM micrograph in back scatter mode of un-etched specimen of composite prepared at the melt temperature of 880 °C, and (b) EDX profile with results of elemental analyses corresponding to the areas marked as 1 in (a).

Standard XRD line profile analyses of the prepared composites and the comparison of the same with that of the base alloy help to identify the developed reinforcement as rhombohedral and monoclinic crystallographic forms of $Al_{13}Fe_4$ phase. Microstructural characterisations establish that stirring of a solid mild steel plate in the super-heated Al-melt resulted in the *in situ* generation of ordered intermetallic of Fe-aluminides (Varin, 2002). The Fe from the mild steel stirrer initially dissolves into the molten-Al until the saturation limit of Fe at the selected melt temperature is reached. Further stirring generates $Al_{13}F_4$ intermetallic particles by the reaction between the super heated liquid-Al and solid mild steel. The generation of some spherical-shape particles in addition to the fibre-shape particles (Fig. 1(a)) is attributed to the presence of rhombohedral as well as monoclinic crystal structures of $Al_{13}F_4$ intermetallic (Chatterjee et al., 2013).

The density values measured by Archimedes principle and the amount of reinforcement estimated via image analyses of composite specimens prepared at different melt temperatures along with those of the base alloy are summarised in Table 1. The density of composite is higher than the base alloy due to the presence of denser intermetallic phases. The density of composite increases with increasing melt temperature indicating the development of higher amount of reinforcing particles. Image analyses results confirm that the volume percent of $Al_{13}F_4$ phase increases monotonically with melt temperature within its investigated range.

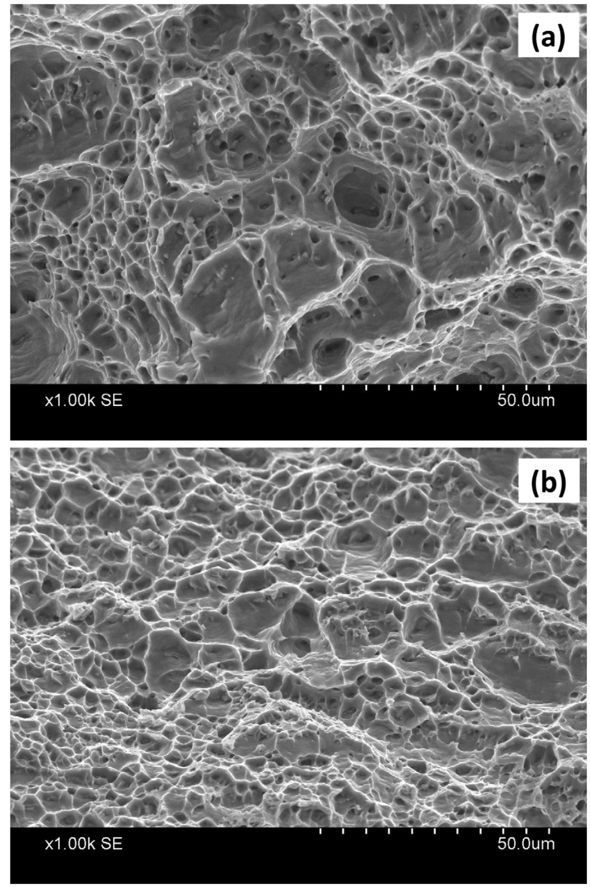

Figure 2 Representative SEM micrographs of tensile fractured surfaces of (a) base alloy and (b) composite specimens.

Mechanical properties

The various mechanical properties of the base alloy and composites are compiled in Table 1. The obtained results demonstrate that the magnitudes of hardness, yield and tensile strength of composite increase significantly with increasing intermetallic content. The variations of parameters related to the ductility and toughness with amount of reinforced are, however, not monotonic in nature. In general, uniform and total elongation values of composites are lower than the base alloy; however, tensile toughness is found to increase with increase in reinforcement upto ~5 vol.% offering best strength-ductility combination. In fact, this composite exhibits higher uniform elongation than that of the base alloy due to higher strain hardening exponent value of the former (Table 1).

Some selected tensile fracture surfaces were examined under SEM in order to understand more than expected ductility of the developed composite. The presence of numerous dimples in fracture surfaces reveals that the mechanism of tensile failure is ductile for both materials (Fig. 2). Fracture surface of the base alloy consists of some very large well-growth voids along with numerous microvoids (Fig. 2(a)), whereas that of the composite exhibits nearly same size of microvoids (Fig. 2(b)). These observations corroborate improved mechanical properties of composite (Table 1). Higher strength-ductility combination of composite is attributed to the better interfacial bond strength between the matrix and the intermetallic particles developed by *in situ* technique (Tjong and Ma, 2000).

Table 1 Summary of microstructure and mechanical properties of base alloy and composites.

Specimens	T_M (°C)	Density (g/cc)	V_F (vol.%)	$HV_{0.5}$ (VHN)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)	*n*	TT (J)
Base alloy	760	2.62 ± 0.01	–	56.0 ± 2.0	62.6	123.1	19.36	27.93	0.198	20.2
Composites	760	2.63 ± 0.01	1.21 ± 0.06	60.1 ± 4.9	77.0	163.1	17.28	23.60	0.196	24.8
	800	2.67 ± 0.01	3.24 ± 0.16	65.8 ± 3.1	90.2	179.3	16.66	23.83	0.187	25.5
	840	2.70 ± 0.01	4.82 ± 0.24	79.6 ± 4.1	95.8	197.5	20.03	26.47	0.274	32.5
	880	2.72 ± 0.01	5.84 ± 0.29	82.7 ± 2.3	97.4	185.2	13.51	17.37	0.196	18.7

T_M: melt temperature; V_F: volume percent of reinforcement (Fe-aluminides); $HV_{0.5}$: vickers hardness at 0.5 kgf load; YS: yield strength; UTS: ultimate tensile strength; UE: uniform elongation; TE: total elongation; *n*: strain hardening exponent; TT: tensile toughness.

Conclusions

Near uniform dispersion of Fe-aluminides particulate reinforced Al-2Mg matrix composite has been developed by stirring of a mild steel plate in the super-heated Al-melt. The intermetallic reinforcement of $Al_{13}Fe_4$ are formed by the *in situ* reaction between the liquid Al and solid mild steel. The amount of reinforced is increased easily by increasing the melt temperature. The developed composite provides higher hardness and improved tensile strength without much loss of ductility as compared to base alloy. Both composite and base alloy exhibit ductile mode of fracture.

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